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HIGH ENERGY OXIDIZERS

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Project NR 093-035

Richmond Research Center

Richmond, California

STAUFFER CHEMICAL COMPANY
Richmond Research Center
Richmond, California

"HIGH ENERGY OXIDIZERS"

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Project NR 093-035

ARPA No. 399-62

OFFICE OF NAVAL RESEARCH
WASHINGTON, D. C.

Quarterly Technical Summary Report
for the Period August 1, 1964 to November 1, 1964

Investigators



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Foreword

This is the third Quarterly Technical Summary Report of the second year's investigation of complexes based on chlorinefluorides. It covers the period from August 1, 1964 to November 1, 1964. The work was conducted at the Richmond Research Center, Richmond, California, of Stauffer Chemical Company, under the sponsorship of the Advanced Research Projects Agency. The work was administered by the Department of the Navy, Office of Naval Research, with Mr. R. L. Hanson serving as Scientific Officer, under ARPA No. 399-62.

Abstract

The difluorohypochlorite anion, i.e., ClF_2^- , a new oxidizer species based on chlorinefluorides, was successfully prepared. Its preparation was achieved in the form of its nitrosyl salt from nitrosylfluoride and chlorinemonofluoride. The complex is a white crystalline solid, which has a 100 mm. dissociation pressure at -51° , and exhibits oxidizing properties. Its composition was established by analysis, molecular weight determination and infrared spectroscopy. It dissociates into the starting materials, NOF and ClF. From a pressure-temperature curve a heat of dissociation of $15.5 \text{ kcal mole}^{-1}$ was obtained. Conductivity measurements proved that the complex is ionic. Low-temperature infrared spectra of the solid complex showed that the structure, $\text{NO}^+\text{ClF}_2^-$, must be assigned to the complex. The ClF_2^- anion belongs to the space group $D_{\infty h}$, and therefore, the most probable structure is a trigonal bipyramid (sp^3d -hybrid) with the chlorine atom at the center, the two fluorine atoms at the apexes and the three free electron-pairs at the remaining corners.

I. Introduction

In the past our efforts were mostly directed towards the structural investigation of the difluorochlorinium cation, i.e., ClF_2^+ , and the combination of this cation with other high energy anions such as nitrate or perchlorate. During the last quarter our work was concentrated mainly on the preparation of the difluorohypochlorite anion, i.e., ClF_2^- , and its structural investigation. A comparison of the structure and properties of the ClF_2^+ cation and the ClF_2^- anion can now be made.

II. Results and Discussion

A. Preparation of $\text{NO}^+\text{ClF}_2^-$

We have recently reported⁽¹⁾ the formation of a white solid compound on passing NOF and ClF at -78° through CFCl_3 solvent. Therefore, the preparation of this compound was repeated in the vacuum line, in order to obtain more information about this complex. The chlorinemonofluoride and the nitrosylfluoride were purified by fractionation and their purity was checked by infrared spectroscopy. Gaseous chlorine-monofluoride showed two absorptions, the P and R branch of the ClF vibration, at 786 and 758 cm^{-1} , respectively, in good agreement with the literature⁽²⁾. Nitrosylfluoride was also in good agreement with the reported spectrum⁽³⁾ except

that we did not find the absorption at 1037 cm^{-1} , which is probably due to a small amount of SiF_4 as an impurity. Our sample contained a small amount of NO_2 as an impurity.

The starting materials were condensed together into a cold trap at -196° without using any solvent. Upon warming and melting, a mild reaction occurred between the two compounds, forming a white solid, the faint yellow color of the liquid chlorinemonofluoride and the light blue color of the liquid NOF disappearing. The excess of one of the compounds can easily be removed in a vacuum at -79° . Preferably, ClF is used in excess, due to its greater volatility (b.p. -100°).

B. Stability and Properties of $\text{NO}^+\text{ClF}_2^-$

Nitrosyldifluorohypochlorite is a white crystalline compound which is not stable at 25° due to its high dissociation pressure. It has a very low solubility in chlorinemonofluoride at -120° and is slightly soluble in NOF at -79° . It reacts violently with water.

A plot of dissociation pressure versus reciprocal temperature (Fig. 1, Table I) for the process:



follows the equation,

$$\log P_{\text{mm}} = -1698 T^{-1} + 9.65$$

in the temperature range -105 to -50° .

PRESSURE - mm

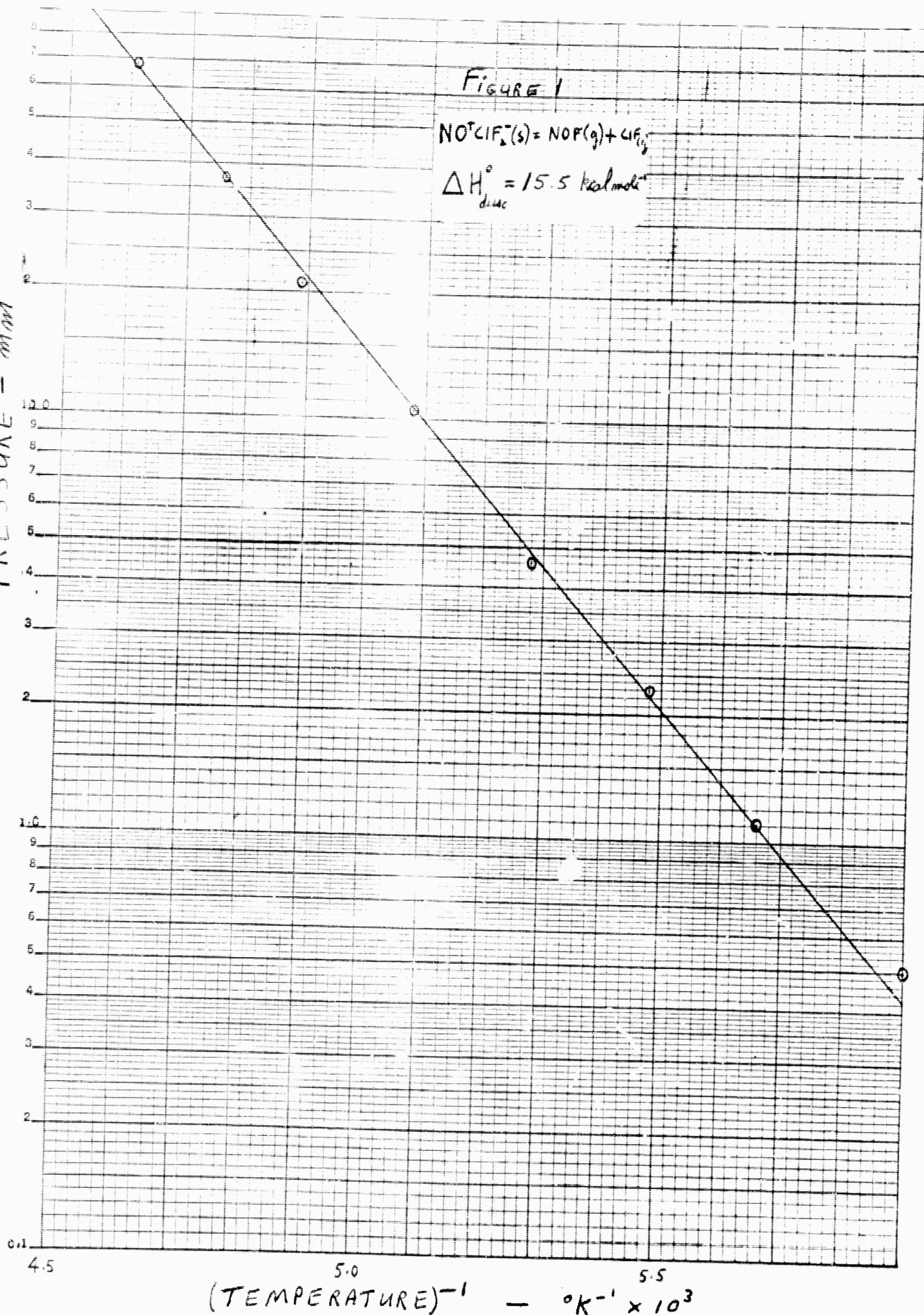


TABLE I

Data for the Heat of Dissociation of $\text{NC}^+\text{ClF}_2^-$

<u>Temperature</u>			<u>Pressure</u>
<u>°C</u>	<u>°K</u>	<u>°K⁻¹ x 10³</u>	<u>mm</u>
-103.7	169.4	5.90	0.5
-96.5	176.6	5.66	1.1
-90.4	182.7	5.48	2.3
-83.8	189.3	5.28	4.6
-76.5	196.6	5.08	10.5
-68.7	204.4	4.89	20.8
-63.0	210.1	4.76	36.8
-55.9	217.2	4.61	68.0
-51.2	221.9	4.51	100.0

By extrapolation, a dissociation pressure of 760 mm was obtained at -20°. The slope of the straight line over this temperature range indicated a heat of dissociation, $\Delta H^\circ_{\text{dissc.}}$, of 15.5 kcal mole⁻¹. Further calculations yielded $\Delta F^\circ_{298} = -2.1$ kcal mole⁻¹, the free energy change, and $\Delta S^\circ_{298} = 44.4$ cal deg⁻¹ mole⁻¹, the entropy change for the process at 25°.

Comparison of the stability of $\text{NO}^+\text{ClF}_2^-$ with that of $\text{NO}^+\text{ClF}_4^-$ (stable at $\sim -5^\circ$ (1)), shows that the ClF_4^- anion is somewhat more stable than the ClF_2^- anion, in good agreement with theoretical expectation. Infrared (Fig. 2) and molecular weight measurements on the gas phase above the complex showed that the complex is completely dissociated (into NOF and ClF) in the gas phase at 25° .

C. Composition of the Complex

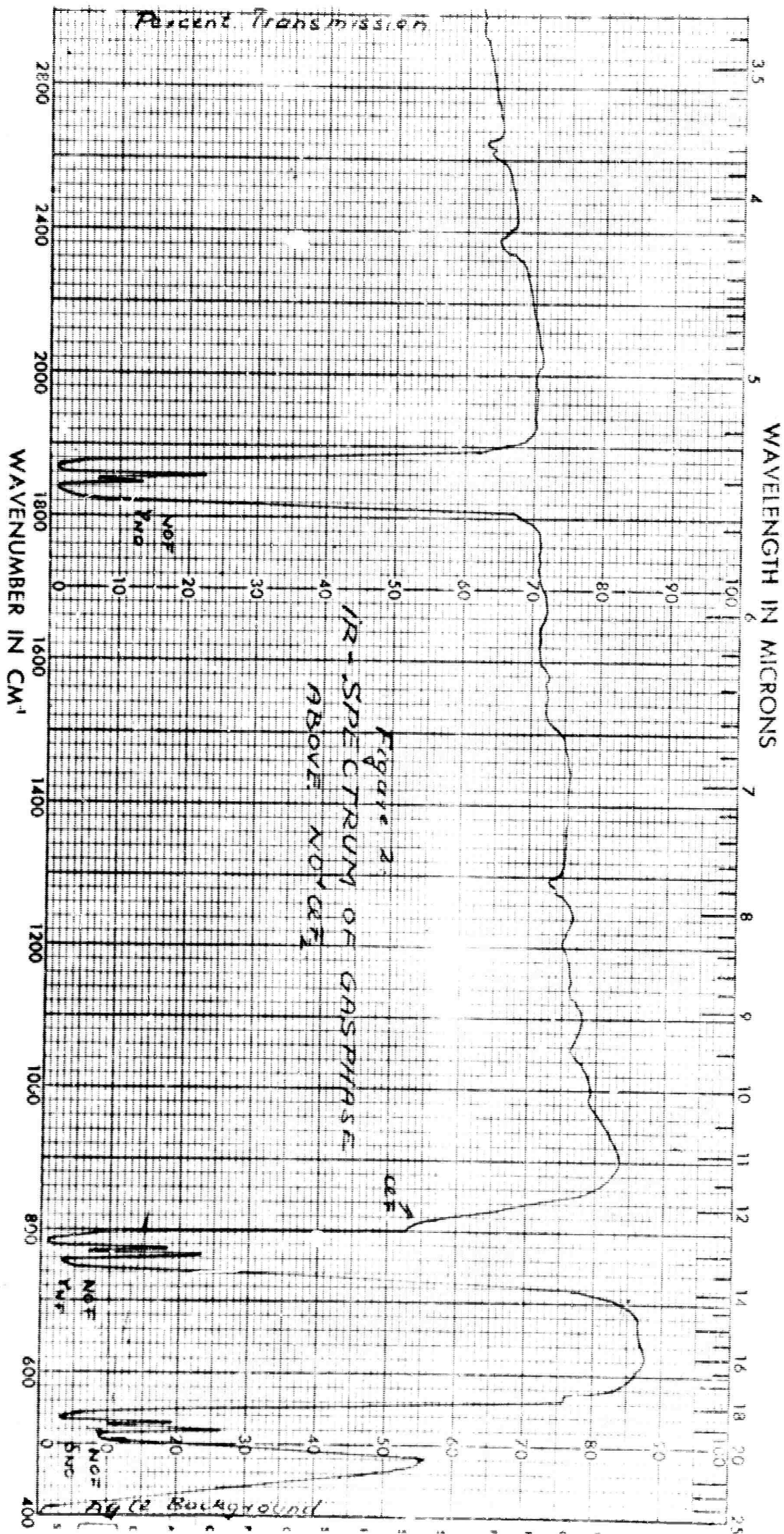
The mole ratio between chlorine monofluoride and nitrosyl fluoride in the complex was established by elemental analysis, molecular weight determination and infrared spectroscopy. All three methods indicate that ClF and NOF combined in a 1:1 mole ratio.

D. Structural Investigation of the Complex

Since NOF and ClF combined in a 1:1 mole ratio, the following three possibilities for the structure of the complex are proposed:

- (1) Ionic complex in form of $\text{Cl}^+\text{NOF}_2^-$
- (2) Ionic complex in form of $\text{NO}^+\text{ClF}_2^-$
- (3) Coordination complex between ClF and NOF, i.e., $\text{ClF}\cdot\text{NOF}$

The decision whether (3) is correct could be made on the basis of conductivity measurements. The choice between (1) and (2) was made on the basis of the infrared spectrum of the solid complex.



Conductivity measurements were made with pure ClF, pure NOF and the complex in NOF solution. Table II shows the results of these measurements.

TABLE II

Conductivity of Complex in NOF Solution

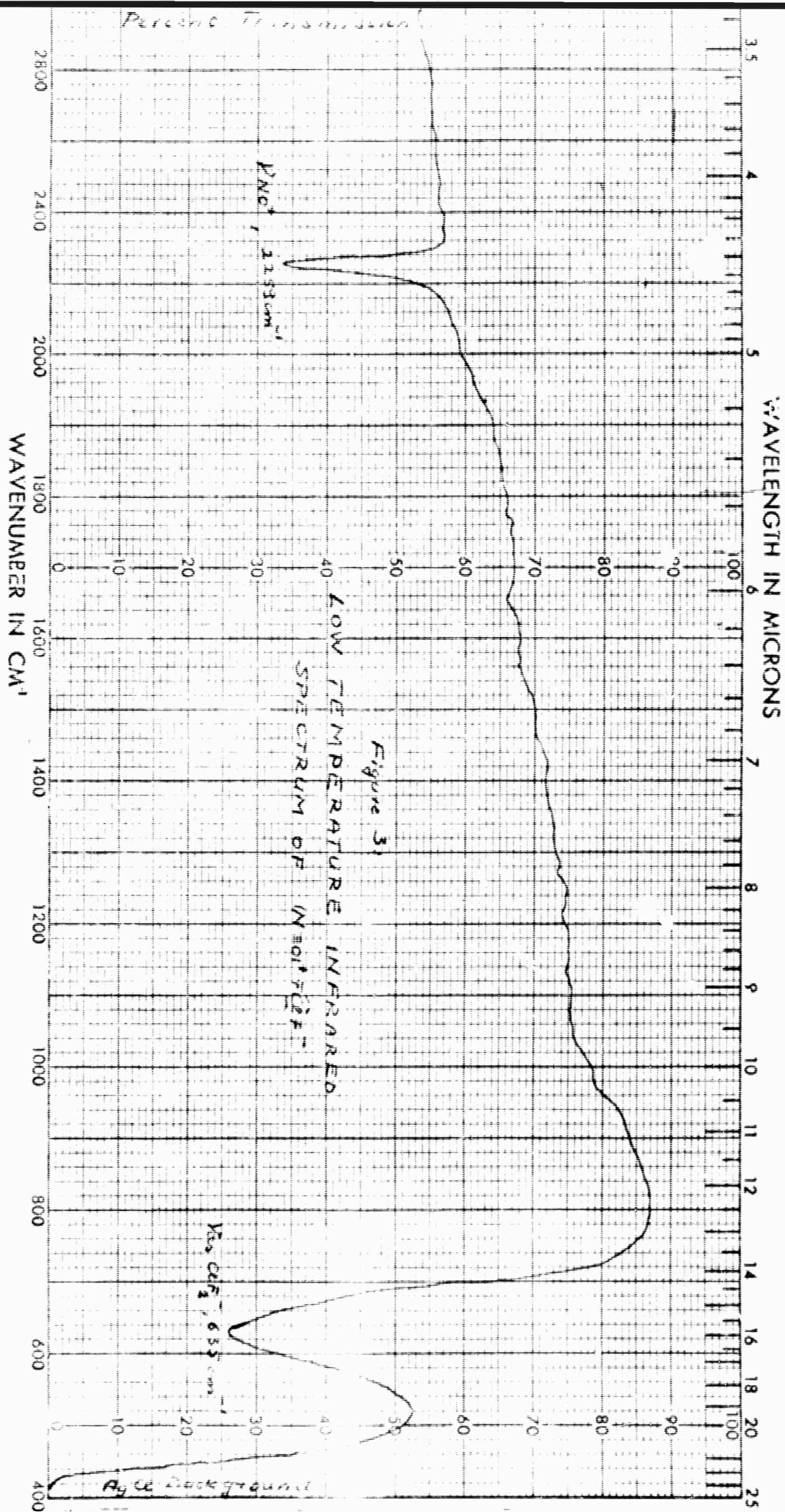
<u>Compound</u>	<u>Temp.</u> <u>(°C)</u>	<u>Molarity</u> <u>(mole l⁻¹)</u>	<u>Specific</u> <u>conductance</u> <u>σ (ohm⁻¹ cm⁻¹)</u>	<u>Equivalent</u> <u>Conductance</u> <u>Λ (ohm⁻¹ cm²)</u>
ClF	-128	--	1.95×10^{-7}	--
NOF	-79	--	5.4×10^{-5}	--
Complex in NOF solution	-79	0.076	3.4×10^{-4}	4.48

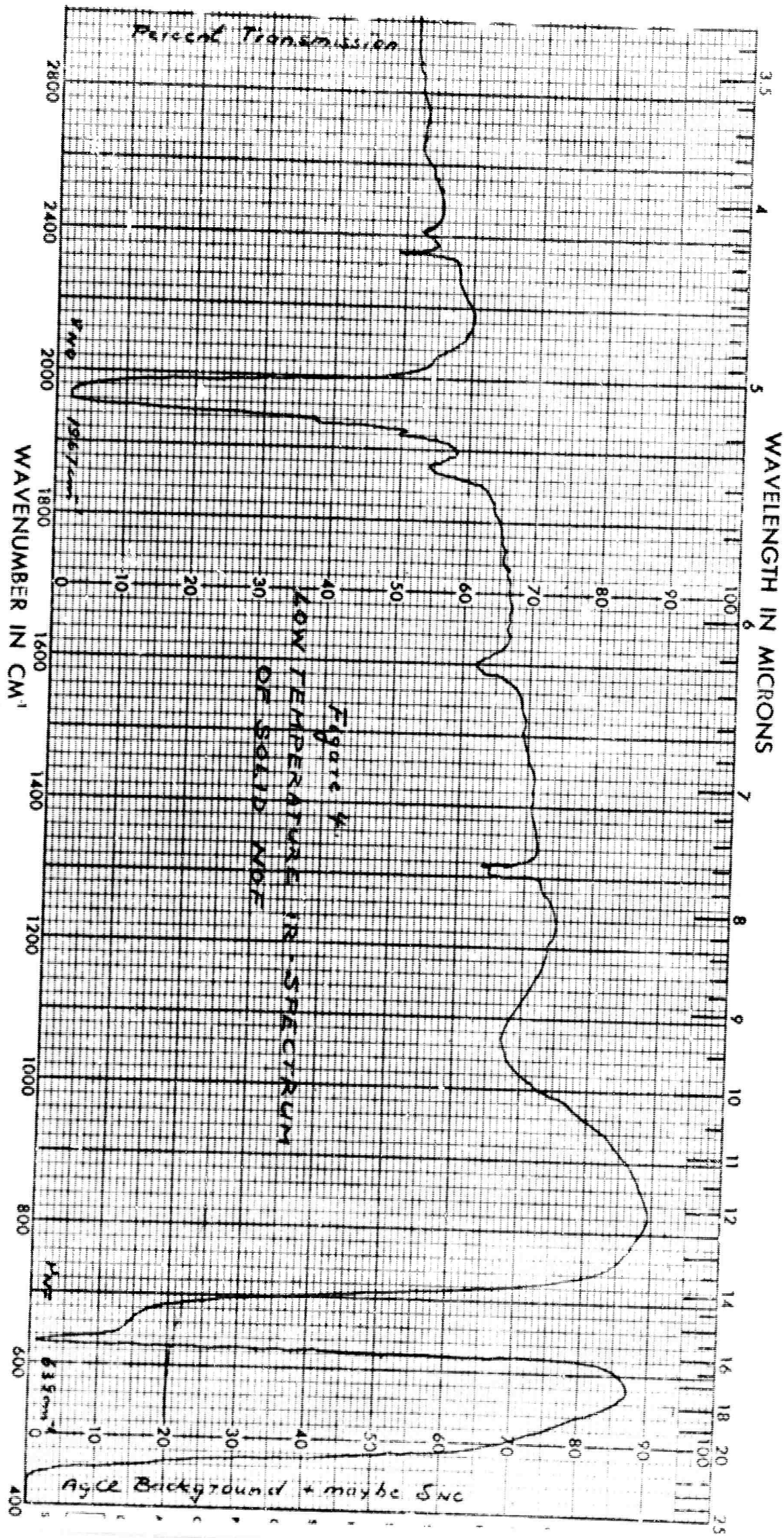
The value of $4.48 \text{ ohm}^{-1} \text{ cm}^2$ obtained for the equivalent conductance of the 1:1 complex in NOF solution compares well with the values obtained for the ionic ClF_2^+ complexes in ClF_3 solution, which were reported⁽⁴⁾ to be in the range of 1.99 to $5.31 \text{ ohm}^{-1} \text{ cm}^2$, depending on the anion. Therefore, structure (3), the coordination complex, is not correct.

Low temperature infrared spectrum: For structure (1), we should expect a total of four infrared active vibrations in the range 4000 to 450 cm^{-1} . For structure (2), we should expect either two (for a linear ClF_2^- anion) or three (for a bent ClF_2^- anion) infrared active fundamentals. In addition,

the position of the highest fundamental, i.e., the N-O bond stretching vibration, will help to differentiate between structures (1) and (2). In the case of structure (1), the N-O has double bond character and should absorb between 1800 and 1900 cm^{-1} . In the case of structure (2) the N-O has triple bond character and should absorb between 2100 and 2350 cm^{-1} (5). Figure 3 shows the infrared spectrum found for the complex. It shows only two absorptions, one at 2259 cm^{-1} and one at 635 cm^{-1} . Therefore, $\text{Cl}^+\text{NOF}_2^-$ is not correct and the $\text{NO}^+\text{ClF}_2^-$ structure must be assigned to the complex. The absorption at 2259 cm^{-1} corresponds to the $:\text{N}\equiv\text{O}:^+$ stretching vibration and 635 cm^{-1} belongs to the asymmetric stretching vibration of the ClF_2^- anion.

The spectra of solid NOF (Fig. 4) and solid ClF were taken, to eliminate the possibility of their presence in the spectrum of the $\text{NO}^+\text{ClF}_2^-$ complex. The spectrum of solid ClF was difficult to obtain, due to its low melting point, but indications are that it shows only one absorption at 748 cm^{-1} . Solid NOF shows considerable changes compared with the spectrum of gaseous NOF. The N=O double bond stretching vibration is shifted 123 cm^{-1} to higher frequency and the N-F stretching is shifted 126 cm^{-1} downwards, indicating a strengthening of the NO bond and weakening of the





NF bond. It does not correspond to an ionization into $\text{NO}^+\text{NOF}_2^-$, but shows that the ionic contributions to the bonds are becoming stronger in the solid state.

When the infrared spectrum of the solid $\text{NO}^+\text{ClF}_2^-$ complex was taken by subliming the complex directly on the AgCl window, a broad peak of medium intensity at about 2000 cm^{-1} was obtained as an impurity, shown by the changes of the intensity ratio of this peak in different runs. However, by preparing the complex from an excess of ClF on the cold window and by pumping off the excess ClF, the peak at 2000 cm^{-1} disappeared completely. If excess ClF was still present together with the complex, six additional peaks between 635 cm^{-1} and 748 cm^{-1} , and two NO absorptions at 2218 and 2280 cm^{-1} were obtained, indicating some interaction between the complex and the ClF. By removal of the ClF, the number of peaks continuously decreased and the only absorption left for the NO part of the complex had an absorption at 2279 cm^{-1} .

Structure of the ClF_2^- anion: The ClF_2^- anion itself could be either linear or bent. If it is linear, it would belong to the space group $D_{\infty h}$ and should show only one infrared active stretching vibration. However, if it belongs to space group C_{2v} (bent molecule), we should expect an asymmetric and symmetric infrared active stretching vibration. Since

only one infrared active stretching at 635 cm^{-1} was observed, the linear structure is probable.

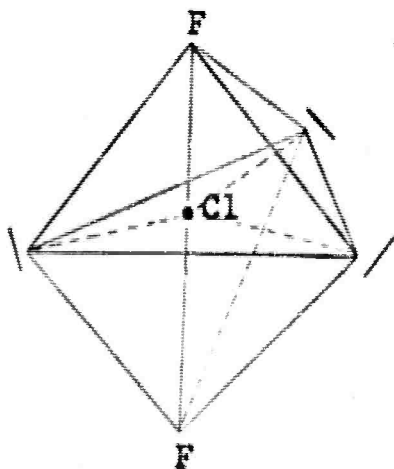
The difluorohypochlorite anion contains three free electron pairs. In order to obtain a linear ClF_2^- anion, we must assume either a sp^3d -hybridization with the two fluorine atoms at the apexes of the trigonal bipyramid, or a sp -hybrid with two free p and one free d electron pairs. Since the sp^3d -hybrid is energetically much more favorable, this structure has the highest probability.

Figure 5 shows a comparison of the ClF_2^+ cation and the ClF_2^- anion, as far as their structure and infrared absorptions are concerned.

FIGURE 5

Comparison of the ClF_2^+ Cation and the ClF_2^- Anion

ClF_2^- anion



space group:

hybridization:

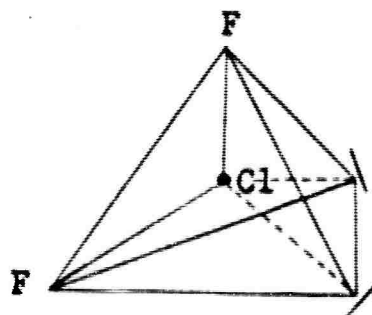
infrared absorptions:

$D_{\infty h}$

sp^3d

$\nu_{\text{as}} \text{ClF}_2^- 635\text{ cm}^{-1}$

ClF_2^+ cation



C_{2v}

sp^3

$\nu_{\text{as}} \text{ClF}_2^+ 558\text{ cm}^{-1}$
 $\nu_{\text{sym.}} \text{ClF}_2^+ 519\text{ cm}^{-1}$

III. Experimental

A. Preparation of $\text{NO}^+\text{ClF}_2^-$

The nitrosyldifluorohypochlorite was prepared from NOF and ClF in the vacuum line. The chlorine monofluoride and NOF were purchased from Ozark-Mahoning Company and purified before use by repeated fractionation in the vacuum line. An all-glass vacuum line and Halocarbon stopcock grease were used and only slight etching of the pyrex glass was observed. The NOF was condensed into a trap, containing a slight excess of ClF, at -196° . Upon warming, the solid complex formed in a mild reaction. The excess of ClF was removed in a vacuum at -79° (dry ice- CHCl_3 - CCl_4 constant temperature bath). The complex was purified by fractionation.

About one gram of the complex was condensed into a pyrex tube which was sealed off under vacuum at -196° and which had a sidearm ending into a capillary tube. The sealed-off tube was quickly weighed on an automatic balance and immediately cooled again with liquid nitrogen. The capillary was broken, the cooling was removed and the complex was slowly hydrolyzed in 5% aqueous NaOH solution.

Analysis: Calc. for F_2ClNO : F, 36.73%; Cl, 34.27%; N, 13.54%

Found: F, 36.0%; Cl, 32.3%; N, 12.9%

The fluoride was determined by titration with thorium

nitrate and sodium alizarin sulfonate as indicator. The chloride was determined by potentiometric titration with AgNO_3 solution after reduction of the positive charged chlorine with sulfite and destruction of the excess sulfite with HNO_3 . The nitrogen was determined by the Kjeldahl method after reduction with Devarda alloy.

B. Molecular Weight Measurements

The complex was prepared as previously described. Using a -45° constant temperature bath (monochlorobenzene) as a moderator, the molecular weight of the gas phase above the complex was determined to an accuracy of better than $\pm 1\%$. The sample of gas at a measured temperature and pressure in a bulb of known volume was weighed and its molecular weight found by subsequent application of the Ideal Gas equation in the form

$$M = \frac{WRT}{PV}$$

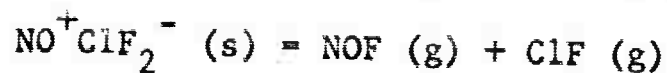
The measurement indicated that the gas phase consisted of NOF and ClF in a 1:1 mole ratio (Found: M, 51.5. Calc. for ClF: M, 54.5; for NOF: M, 49.0; Average assuming 1:1 mole ratio: M, 51.7; Calc. for ClF_2NO : M, 103.5).

C. Dissociation Pressure Measurements

The complex was prepared directly in a LeRoy still⁽⁶⁾.

Temperatures were measured using a copper-constantan thermocouple (standard at 0°) connected to a Honeywell potentiometer Model 2733. Pressures were measured using a soft glass (flint type) spoon gauge as a null indicator in order to prevent direct contact of any corrosive gases with mercury. To be certain that an equilibrium existed at each temperature reading, a sample of the gas above the solid complex was pumped off and a constant pressure was re-established. True equilibrium existed at a given temperature if the pressure before and after the pumping off procedure were identical. Pressure-temperature measurements were made until the complex decomposed irreversibly, as evident by the failure to observe identical pressures before and after the pumping off operation and by the formation of a brown colored gas at pressures greater than 100 mm. (probably due to etching of the glass). Equilibrium pressures were approached from above as well as below a given temperature.

In general, for a heterogeneous equilibrium, as



the heat of dissociation is given by the van't Hoff reaction isobar⁽⁷⁾,

$$\Delta H^\circ = -R \frac{\Delta \ln K_p}{\Delta T^{-1}}$$

where K_p is the equilibrium constant. The slope of the linear $\log K_p$ vs. T^{-1} plot gives ΔH° directly. We obtained a $\log P_{\text{mm}}$ vs. T^{-1} plot, the slope of which gave ΔH° if the relation, $K_p = \frac{1}{2} P^2$, was used. In making this calculation it was assumed that the complex was fully dissociated in the gas phase and that the solid complex had no pressure, i.e., it had unit activity.

The method gave $\Delta H^\circ_{\text{dissc.}}$ to an estimated accuracy of ± 0.3 kcal mole $^{-1}$. This was based on the following possible experimental errors:

temperature, T ($^\circ\text{C}$), ± 0.2

pressure, P (mm), ± 0.1

and recalculating $\Delta H^\circ_{\text{dissc.}}$ using pressure-temperature errors at the two points $P = 20$ mm. and $p = 2$ mm., to produce a maximum and a minimum $\Delta H^\circ_{\text{dissc.}}$. The human error in reading the slope of the straight line was less than ± 0.05 kcal mole $^{-1}$, as five different co-workers read the slope within this accuracy.

The equation,

$$\log P_{\text{mm}} = -1698 T^{-1} + 9.65$$

represents the dissociation pressure data in the temperature range -105 to -50° . Using $K_p = \frac{1}{2} P^2$, the equation,

$$\log K_{p(\text{atm})} = \frac{-3396}{T} + 12.94$$

was obtained. The free energy charge, ΔF° , for the dissociation process was calculated using

$$\Delta F_T^\circ = -RT \ln K_p(\text{atm})$$

Thus,

$$\Delta F_{298}^\circ = -2.1 \text{ kcal mole}^{-1}$$

Finally, the entropy charge, ΔS° , for this process was found from

$$\Delta S_T^\circ = \frac{\Delta H^\circ - \Delta F_T^\circ}{T}$$

Thus,

$$\Delta S_{298}^\circ = 44.4 \text{ cal deg}^{-1} \text{ mole}^{-1}$$

D. Conductivity Measurements

The conductivity measurements were carried out in a pyrex cell, connected to the vacuum line. The cell could be closed by a stopcock. It contained two platinized Platinum electrodes with platinum leads. The glass-metal seal in the cap of the cell was made by using tungsten rods which were spot welded to the platinum leads. The tungsten rods were sealed over their complete length inside of the cell with glass, in order to protect them against chemical attack. The resistance was measured with a Heathkit decade resistance, Model IN-11 and a Heathkit capacitor checker, Model IT-11. The ClF, NOF and the complex were repeatedly fractionated in

the vacuum line before use. The cell constant of the conductivity cell was determined with 0.01N HCl and found to be 12.4. The results of the measurements are given in Table I. Attempts to determine the conductivity of the complex in ClF as solvent at -128° were unsuccessful and no increase in the conductivity could be observed. This is probably due to the extremely low solubility of the complex in the ClF under these conditions.

The concentration of the complex in the NOF was determined by measuring the molar ratio between NOF and ClF volumetrically in the gas phase and converting the so obtained molality into molarity by using the equation given in the literature⁽⁸⁾, for the density of liquid NOF.

E. Infrared Spectroscopic Investigations

All infrared spectra were taken with a Beckman IR-9 in the range 4000 to 450 cm^{-1} . Pyrex glass cells equipped with AgCl windows were used. The AgCl windows were sealed with a thin film of Halocarbon stopcock grease to the glass body. For the low-temperature spectra, a modified cell with an internal AgCl window, which was kept cold with liquid N_2 , was used. The cell was described in one of our earlier reports⁽⁹⁾.

In order to establish the 1:1 ratio of the complex by

infrared, calibrations were made with pure NOF and ClF at different pressures. The vapor phase above the complex showed at a total pressure of 200 mm. in a 1 cm. gas cell exactly the same intensities for NOF, as a sample of NOF alone showed at 100 mm. pressure.

vember 24, 1964

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